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05- 05-03

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J1036 U.S. PTO
60/467311

PROVISIONAL APPLICATION COVER SHEET

This is a request for filing a PROVISIONAL APPLICATION FOR PATENT under 37 CFR 1.53 (b) (2).

Docket Number	135143-1 60SD	Type a plus sign (+) inside this box→	+

INVENTOR(s) APPLICANT(s)

LAST NAME	FIRST NAME	MIDDLE INITIAL	RESIDENCE (CITY AND EITHER STATE OR FOREIGN COUNTRY)
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TITLE OF THE INVENTION (280 characters max)

POLYCRYSTALLINE DIAMOND TOOLS AND METHOD OF MAKING THEREOF

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ENCLOSED APPLICATION PARTS (check all that apply)

[X] Specification	Number of pages [12]	[ ] Small Entity Statement
[ ] Drawing(s)	Number of sheets [5]	[ ] Other (specify) [assignment]

METHOD OF PAYMENT FOR FILING FEES FOR THIS PROVISIONAL APPLICATION FOR PATENT

[ ] A check or money order is enclosed to cover the Provisional filing fees	PROVISIONAL FILING FEE AMOUNT ($)	$160.00
[X] The Assistant Commissioner is hereby authorized to charge filing fees and credit Deposit Account Number [07-0860]		

The invention was made by an agency of the United States Government or under a contract with an agency of the United States Government

[X] No
[ ] Yes, the name of the U.S. Government agency and the Government contract number are:

Respectfully submitted,

SIGNATURE ____________ DATE 5-1, 2003

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PHONE NUMBER (413)-448-4664

HTP/jar

Attorney's Docket No. 135143-1 60SD Patent

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of: Shan Wan

FOR: **POLYCRYSTALLINE DIAMOND TOOLS AND METHOD OF MAKING THEREOF**

Box Provisional Patent Application
Assistant Commissioner for Patents
Washington, D.C. 20231

COVER SHEET FOR FILING PROVISIONAL APPLICATION
(37) C.F.R. §1.51 (2) (i))

Sir:

Enclosed is a Provisional Application for filing. Detailed Information is set forth on page two of this cover sheet.

CERTIFICATION UNDER 37 CFR 1.10

I hereby certify that this correspondence and the documents referred to as attached therein are being deposited with the United States Postal Service on 5/2/, 2003, in an envelope as "EXPRESS MAIL POST OFFICE TO ADDRESSEE" service under 37 C.F.R. 1.10, Mailing Label Number EL 854526397US addressed to the: Assistant Commissioner for Patents, Washington, D.C. 20231.

Date: May 2, 2003

Karen Hayden
Karen Hayden

Provisional Application

POLYCRYSTALLINE DIAMOND TOOLS AND METHOD OF MAKING THEREOF

[001] The present invention relates to a PCD tools and methods of making thereof.

BACKGROUND OF THE INVENTION

[002] Polycrystalline diamond tools are extensively used in drilling, cutting, and machining applications. Extensive efforts have been made to improve the abrasion resistance and impact resistance. Conventionally, large diamond grain size leads to high impact resistance, but relatively low abrasion resistance for drilling cutter. Fine diamond grain size is preferred for better abrasion resistance. It has been a challenging problem to achieve both high impact resistance and abrasion resistance for polycrystalline drilling cutters.

[003] Grain size dependence of the performance of polycrystalline diamond drilling cutters has been extensively investigated in the prior art. Different from normal ceramics such as alumina, silicon carbide, whose fracture toughness increases with decreasing of the grain size, fracture toughness of PCD, which determines the impact resistance of the cutter, actually decreases with finer diamond grain size as disclosed in Miess, D. and Rai, G., Fracture Toughness and Thermal Resistance of PDC, *Materials Science and Engineering*, A29,270-276, 1996.

[004] Therefore, to avoid severe diamond table failure such as delamination and spall in high impact drilling applications, usually a coarse grain size microstructure is desired. However, the abrasion resistance is sacrificed with the large grain size. The lifetime of the cutter is limited because of the fast wear of the diamond table. Extensive efforts have been made to avoid this dilemma by varying the diamond table configuration. US Patent No. 4,311,490 describes a non-uniform diamond table configuration including upper fine grain layer and lower coarse grain layer. US Patent No. 4,604,106 proposes a PCD compact comprising a transition layer with diamond – carbide composite between normal carbide substrate and working PCD layer.

[005] EP Patent Application No. 1190791 describes a non-uniform microstructure with gradient distribution of catalyzing materials. With these non-uniform microstructures, the

fracture toughness of the portion of diamond table close to supporting substrates can be improved. However, the top portion of diamond table is still very brittle and tends to fail under high impact. There is still a need for a better microstructure to improve the cutter performances.

[006] In the high-pressure high-temperature sintering process, the grain size of PCD is mainly determined by the starting diamond particle size. Therefore, by controlling the starting particle size, it is possible to control the final microstructure. The impact strength of the PCD body is greatly dependent on the diamond-to-diamond bonding. High extent of diamond-to-diamond bonding is preferred to achieve better performance. This can be accomplished by intentionally increasing the starting powder packing density. Theoretically, the highest relative density of single size sphere packing body is only 0.74, and the highest relative density of bi-modal powder packing body, which containing two type of single size particles is 0.93. Particle shape also greatly affects the packing of the green body. Irregular particle shape usually leads to lower packing density than that of perfect spheres. Applicants are not aware of prior disclosure on shape control of the starting diamond powder to improve the packing density.

[007] US Patent No. 5766394 describes some examples made with a particle size distribution including three different average particle sizes. Their particle size distribution showed a continuous size variation instead of mixing of two types of tightly controlled single size powders. US Patent No. 6261329 proposes a diamond sintered body consisted of particles with sizes ranging from 0.1 micron to 70 microns. These powders have continuous particles size distribution, and not made from two types distinct particles sizes. US Patent Nos. 5468268 and 5505748 also suggest a tri-modal powder mixture to make PCD compact. Based on the example provided by US Patent No. 5505748, the calculated relative density of packing body will be between 0.66-0.72 using the extended Westman model (See Westman, A. E. R., and Hugill, H. R., The Packing of particles, *J. Am. Ceram. Soc.*, 13[10], 767-769, 1930). No relationship among powder size distribution, particle shape, packing density and final performance of cutting tool is disclosed. Actually, a higher packing density can be achieved by just using bi-modal powder mixture, that is, a powder mixture with two different average particle sizes.

[008] US Patent No. 5855996 described a mixture of submicron sized diamond particles and large sized particles. Only average size of the powder is defined in this disclosure. The size distribution of each type of powder is not specified. Even with same average particle size, different size distribution can lead to very different packing density and final performance. Again, no shape control is disclosed in all of these prior patents.

[009] Applicants have surprisingly found an optimum powder mixture with shape and volume fraction controlled fine particles and coarse particles, overcoming the disadvantages of the single size diamond grain microstructure and improving the overall performance of polycrystalline diamond (PCD) drilling cutters in terms of abrasion resistance and impact resistance.

SUMMARY OF THE INVENTION

[010] The invention relates to cutting elements, comprising sintered polycrystalline diamond or CBN starting from a feed of bi-modal powder mixture of two different types of single size particles.

[011] The invention further relates to improve the impact resistance and abrasion resistance of cutting elements by the use of PCD or cubic boron nitride crystals starting from a feed of bi-modal powder mixture of two different types of single size particles.

BRIEF DESCRIPTION OF THE DRAWINGS

[012] Fig. 1 is a graph illustrating one embodiment of the invention, with the packing density as a function of particle aspect ratio for single size diamond particles.

[013] Fig. 2 1 is a graph showing the calculated bi-modal diamond particle packing densities as a function of fine particle volume fraction with various particle size ratio *r*.

[014] Fig. 3 is a graph showing the bi-modal powder packing density as a function of fine particle volume fraction.

[015] Fig. 4 1 is a graph showing the particle size distribution of the bi-modal powder mixture used in one embodiment of the invention, cutter C.

[016] Fig. 5 is a graph comparing the performance between the bi-modal feed cutters of one embodiment of the invention, and the mono-modal feed cutter of the prior art.

DETAILED DESCRIPTION OF THE INVENTION

[017] The invention relates to tools and / or cutting elements for machine wear materials, including rotary drill bits for use in drilling or coring holes in subsurface formations. The invention may be applied to a number of different kinds of drill bits, including drag bits, roller cone bits and percussion bits.

[018] By way of example, the invention will be primarily described in relation to a cutting element which comprises a preform element, often in the form of a circular tablet, including a cutting table of superhard material having a front cutting face, a peripheral surface, and a rear face, the rear face of the cutting table being bonded to a substrate of material which is less hard than the superhard material.

[019] The cutting table usually comprises polycrystalline diamond ("PCD") crystals, although other superhard materials are available and may be used, such as cubic boron nitride. The substrate of less hard material is often formed from cemented tungsten carbide, and the cutting table and substrate are bonded together during formation of the cutting element in a high pressure, high temperature forming press. This forming process is well known and will not be described here. The preform cutting element may be directly mounted on the bit body or may be bonded to a carrier disc, for example also of cemented tungsten carbide, the carrier being in turn received in a socket in the bit body. The bit body may be machined from metal, usually steel, or may be formed from an infiltrated tungsten carbide matrix by a powder metallurgy process.

[020] In one embodiment, the substrate may be formed by joining together two or more disparate carbide discs in the HTHP sintering process to form the PDC cutter. The carbide discs may vary from each other in binder content, carbide grain size, or carbide alloy content. In another embodiment, the carbide discs may be selected and arranged, therefore, to produce a gradient of materials content in the substrate which modifies and provides the properties for the cutting table.

[021] The diamond clusters forming the cutting table are produced by a method which provides a source of carbon and a plurality of growth center particles, each growth center particle comprising a bonded mass of constituent particles, producing a reaction mass by bringing the carbon source and the growth center particles into contact with a solvent/catalyst, subjecting the reaction mass to conditions of elevated temperature and pressure suitable for crystal growth and recovering a plurality of the diamond clusters, as discrete entities, from the reaction mass. The carbon source may be graphite, HPHT (high pressure high temperature) synthetic diamond, chemical vapor deposited (CVD) diamond or natural diamond, or a combination of two or more thereof or other carbon sources known in the art.

[022] Diamond crystals are commercially available from a number of suppliers including, for example, General Electric Company, DeBeers Industrial Diamond Division, or Dennis Tool Company.

[023] The dependence of relative density of a diamond powder packing body on particle shape is determined experimentally. As shown in Fig.1, for single size diamond particles, the particle aspect ratio near 1.0 leads to higher packing density. Therefore, it is desired to have blocky particles with aspect ratio close to 1.0 to achieve high green body packing density.

[024] The diamond crystals in the present invention have relatively large aspect ratios. In one embodiment of the invention, the diamond crystals have largely well defined cubo-octahedral shapes. In a second embodiment, the crystals may have a large aspect ratio in various shapes, including ellipsoids. In a third embodiment, the crystals are essentially two dimensional such as laminas and/or flakes. In yet another embodiment, the crystals are essentially one dimensional, e.g. rods, fibers and/or needles.

[025] The packing model specifically for diamond powder mixture is developed based on the initial single size particle packing densities. It shows the high green body packing density can be obtained by uniformly mixing two types of particles with controlled particle size and shape distribution. Fig. 2 shows the relative density of a diamond powder

packing body calculated from the model as a function of volume fraction of two different size particles and their particle size ratio *r*, where *r* = fine particle size/ coarse particle size.

[026] As shown in Fig.2, the binary powder mixture packing density is mainly dependent on these factors: initial packing density for each single size particles, which is determined by the particle shape, particle size ratio between two different size particles, and volume fraction of each single size powder. It indicates that a lower particle size ratio usually leads to a higher packing density. It means that larger size difference is preferred for achieving closer packing. On the other hand, the volume fraction greatly affects the packing density. It can be seen that for a fixed particle size ratio, a bi-modal powder mixture with around 70% coarse particles and around 30% fine particle has the highest packing density. With higher green body packing density, the powders are less crushed under high-temperature and high-pressure process. This will contribute to higher impact resistance.

[027] EXAMPLE. The examples below are merely representative of the work that contributes to the teaching of the present invention, and the present invention is not to be restricted by the examples that follow.

[028] The following tests may be conducted to measure the impact resistance and abrasion resistance properties of the cutting tools of the present invention and the comparative prior art samples.

[029] *Abrasion Resistance Test:* In this test to measure the abrasion resistance, with each piece having a carbide chamfer of greater than about 0.2 mm, less than 1.0 mm radial or 45° on the locating base. First, a granite log is fitted to a lathe. The cutter (sharp edge) is mounted into a steel support. The cutter (rake angle 15 degrees) is run across the rotating log. The size of the wear land on the cutter is measured after each pass of the log. The volume of material removed from the log is measured. The values are plotted against each other giving the abrasion resistance of the cutter.

[030] *Parkson Mill Test:* This test is to estimate the performance of the cutter on a chamfer piece, with each piece having a carbide chamfer of greater than about 0.2 mm, less than 1.0 mm radial or 45° on the locating base. The Diamond table has a 0.012 inch chamfer.

In this test, the cutter (chamfered edge) sample is mounted in a steel holder. The cutter is rotated and cuts in an interrupted fashion and transverse distance of 0.15 inch through a granite work piece. The test is stopped when the diamond table fails, and the number of impacts (entries into the log) counted.

[031] In the examples, two types of PCD diamond particles commercially available from General Electric Company of Worthington, OH, having particles with average particle size of 85 micron and 20 micron are mixed uniformly. The experimental packing density of the powder mixture is shown in Fig. 3. It can be seen the bi-modal powders can increase the packing density by up to 20% compared to single particle size powder. The particle size distribution of a typical bi-modal powder mixture is shown in Fig. 4. The tool is sintered by normal high-temperature high-pressure process.

[032] The abrasion resistance of the tool is measured by granite-log wear test (Abrasion Resistance Test as described above). The test sample has a cylinder shape with a diameter of 13mm and a height of 13mm. The diamond table thickness is 2.5mm. The cutting edge of test part is kept up-sharp without chamfering. Test is performed on an 8-12 inches diameter granite-log installed on a lathe. The rotation speed of granite log is controlled with constant surface moving speed: 300 SFPM. The cutting tool has 15 degrees of rake angle and moves parallel to the center-line of the log. Cutting depth of the tool into the granite log is 0.01 inch. The cross-feed is 1.5 inch/min. The wear land area is measured every 2 minutes cutting. Test is stopped after 18 minutes. The abrasion resistance is calculated as final volume ($inch^3$) removed by the tool divided by the final wear land area ($inch^2$).

[033] The impact resistance is characterized by interrupting impact test performed on Parkson Mill test machine (Test is as described above). Samples have the same geometry as those for abrasion test. Each sample has a 0.012 inch, 45 degrees chamfer on the test edge. The sample is held by a tool holder spinning at 320 RPM. The tool cuts into a granite block with a depth of 0.15 inch and 15 degrees rake angle. Each granite block is 16 inches long and moves along the cutting plane with a speed of 2.1 inch/min. Pass is complete when downward stroke of the tool has cleared the block. After each pass, the granite block is moved back to the starting point and moved toward the cutting tool to establish a new 0.15 inch

cutting depth. The impact resistance is measured by the number of the interrupting hits on the tool before the tool fails. Tool failure is defined as when the diamond table has been worn to the point that the WC substrate is exposed.

[034] With the optimized bi-modal powder mixture, the performance of the PCD cutting tool is surprisingly improved. The impact resistance number in Table 1 represents the overall hit number on the cutter before it loses cutting efficiency. The abrasion resistance number in Table 1 represents the tool efficiency defined as the ratio of the removed granite materials volume over the wear land area of the cutter. Higher tool efficiency means better abrasion resistance.

[035] Cutter A and B are comparable / standard cutters made of traditional single size particles commercially available from various sources, including General Electric Company of Worthington, OH. A is made of coarse particles with average size of 85 micron and average particle aspect ratio of 0.81. B is made of fine particles with average particle size of 20 micron and average particle aspect ratio of 0.67. The particle size distributions for both powders were controlled so that the standard deviations of particle size distributions are less than 0.3d, where d is average particle size. Cutter C is made from the bi-modal feeds of the present invention by mixing the coarse particles used in Cutter A and fine particles used in cutter B.

[036] Table 1 shows the impact resistance and abrasion resistance of three different cutters. As shown in Table 1, compared to standard single coarse particle size cutter A, cutter C with bi-modal maintains high impact resistance and has three times higher abrasion resistance. Compared to standard single fine particle size cutter B, cutter C with bi-modal has 50% higher impact resistance and 20% higher abrasion resistance.

[037] Fig. 5 summarizes the performance comparison between bi-modal cutters and mono-modal cutters. Apparently, the conventional mono-modal cutters locate in the left-lower part of the performance graph. The performance zone of conventional cutters is below the dash line which can be defined as an equation: Number of impact resistance + Number of abrasion resistance < 19000. The high performance bi-modal cutters located in the right-upper part of the performance graph, which is above the dash line.

Table 1 Summary of impact resistance and abrasion resistance of cutters made of traditional feeds and bi-modal feeds.

	Coarse Particle vol%	Fine Particle vol%	Particle Size Ratio	Particle Aspect Ratio	Impact Resistance	Abrasion Resistance
A: Standard Cutter with single coarse size particle	100%	0	–	0.81	15029	2731
B: Standard Cutter with single fine size particle	0	100%	–	0.67	10500	7500
C: Bi-modal Cutter Example 1	40%	60%	0.22	–	15600	10048

[038] While the invention has been described with reference to a preferred embodiment, those skilled in the art will understand that various changes may be made and equivalents may be substituted for elements thereof without departing from the scope of the invention.

CLAIMS

We claim:

1. A tool insert comprising a continuous abrasive layer having a periphery forming a cutting surface said abrasive layer and/or a support layer on said substrate.

2. The tool insert of claim 1, wherein said the top abrasive layer is made of superhard materials including polycrystalline diamond or/and cubic boron nitride.

3. The tool insert of claim 2, wherein said the tool abrasion resistance and impact resistance have the relationship defined as: value of impact resistance + value of abrasion resistance ≥ 19000.

4. The tool insert of claim 3, wherein said the polycrystalline diamond or CBN are sintered with high pressure high temperature process starting from a bi-modal powder mixture.

5. The tool insert of claim 4, wherein said the bi-modal powder mixture includes two different types of single size particles.

6. The tool insert of claim 5, wherein the average size ratio of fine particle over coarse particle is between 0.02 and 0.75.

7. The tool insert of claim 6, average size ratio of fine particle over coarse particle is between 0.05 and 0.5.

8. The tool insert of claim 7, wherein the average size ratio of fine particle over coarse particle is between 0.1 and 0.5.

9. The tool insert of claim 5, wherein the standard deviation of particle size distribution of each type of single size particles is smaller than 0.6d, where d is the average particle size.

10. The tool insert of claim 9, wherein the standard deviation of particle size distribution of particle size distribution of each type of single size particles is smaller than 0.5d, where d is the average particle size.

11. The tool insert of claim 10, wherein the standard deviation of particle size distribution of the standard deviation of particle size distribution of each type of single size particles is smaller than 0.4d, where d is the average particle size.

12. The tool insert of claim 5, wherein the diamond crystals have an average aspect ratio of particles of greater than about 0.2.

13. The tool insert of claim 5, wherein the diamond crystals have an average aspect ratio of particles of greater than about 0.3.

14. The tool insert of claim 5, wherein the diamond crystals have an average aspect ratio of particles of greater than about 0.4.

15. A tool insert as defined in claim 5, wherein the volume fraction of small size particles is between 5% to 90%, and the volume fraction of large size particles is between 10% to 80%.

16. A tool insert as defined in claim 5, wherein the volume fraction of small size particles is between 10% to 90%, and the volume fraction of large size particles is between 10% to 70%.

17. A tool insert as defined in claim 5, wherein the volume fraction of small size particles is between 15% to 90%, and the volume fraction of large size particles is between 10% to 60%.

POLYCRYSTALLINE DIAMOND TOOLS AND METHOD OF MAKING THEREOF

ABSTRACT:

The invention relates to cutting elements having improved impact resistance and abrasion resistance properties, comprising sintered polycrystalline diamond or CBN starting from a feed of bi-modal powder mixture of two different types of single size particles. The invention further relates to a method for making cutting elements having improved impact resistance and abrasion resistance properties, by the use of PCD or cubic boron nitride crystals starting from a feed of bi-modal powder mixture of two different types of single size particles.

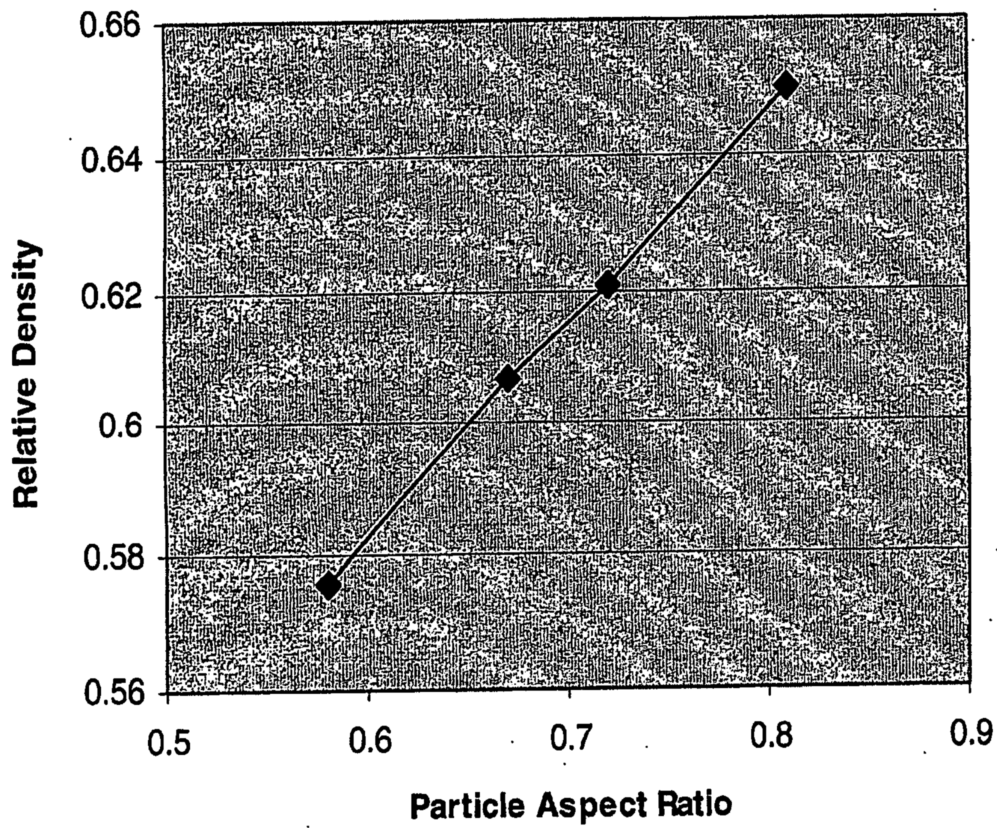

0.66
0.64
0.62
0.6
0.58
0.56
Relative Density
0.5
0.6
0.7
0.8
0.9
Particle Aspect Ratio

FIGURE 1

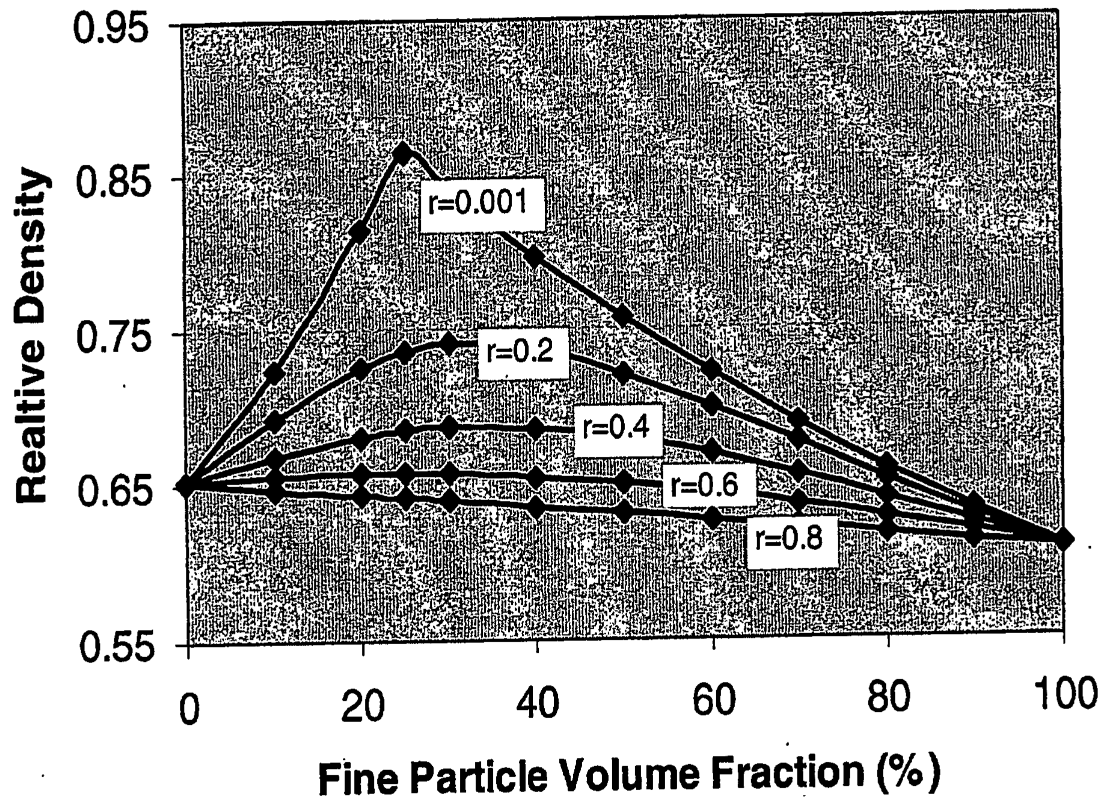

0.95
0.85
0.75
0.65
0.55
Realtive Density
0
20
40
60
80
100
Fine Particle Volume Fraction (%)
r=0.001
r=0.2
r=0.4
r=0.6
r=0.8

FIGURE 2

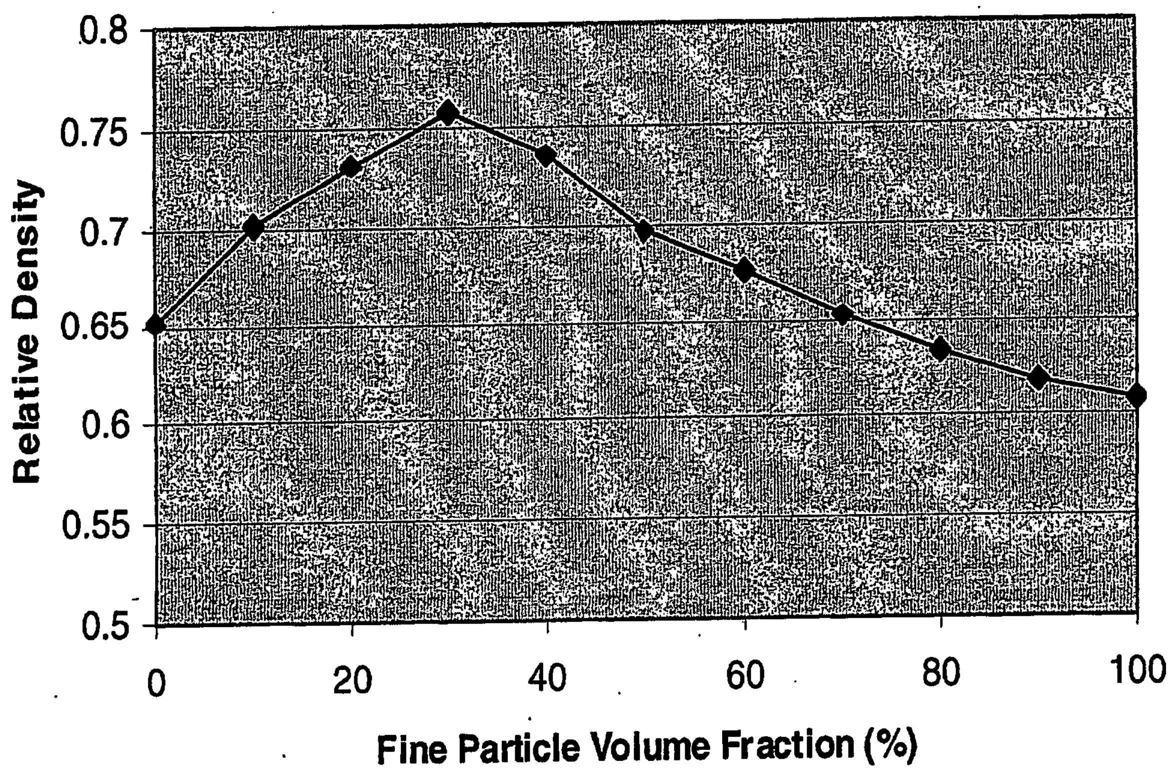
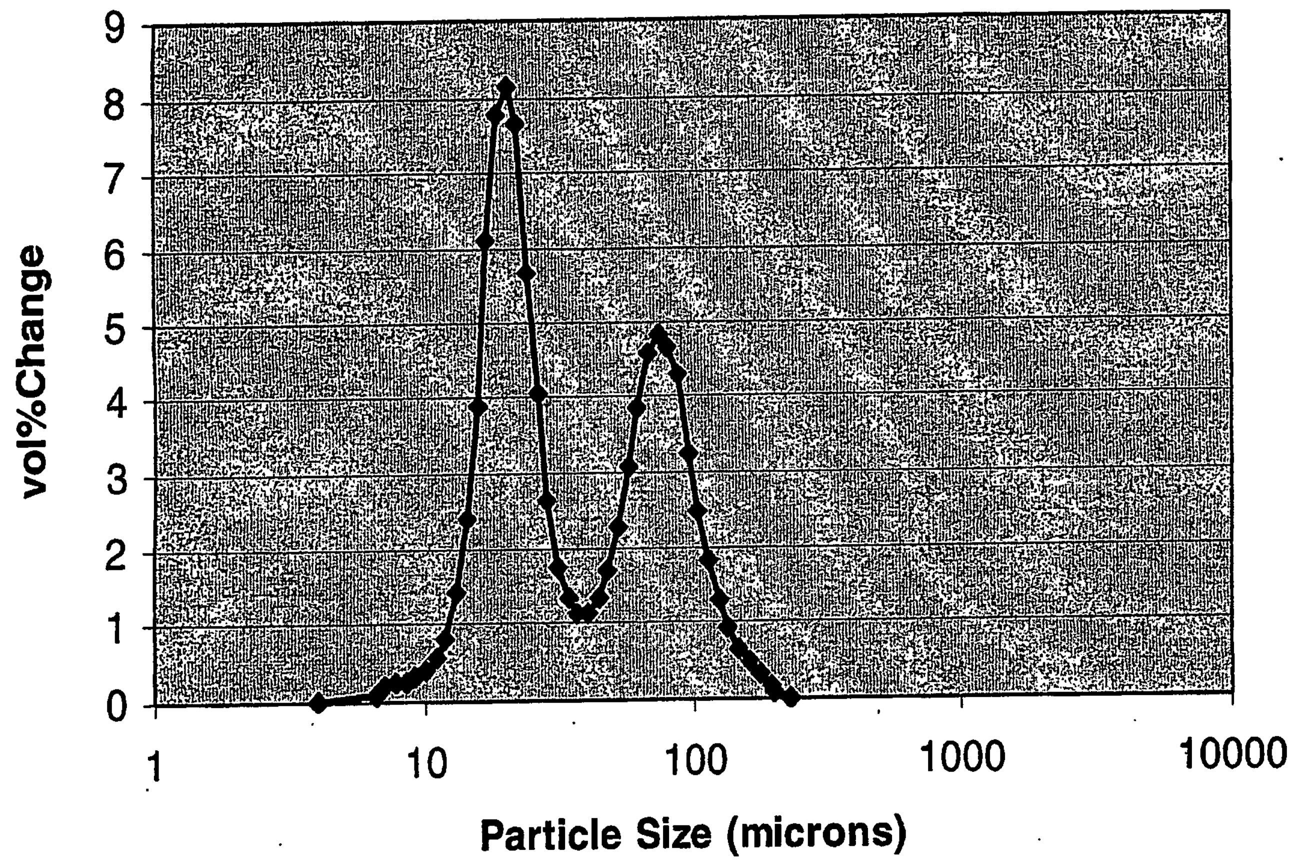

0.8
0.75
0.7
0.65
0.6
0.55
0.5
Relative Density
0
20
40
60
80
100
Fine Particle Volume Fraction (%)

FIGURE 3



vol%Change
0
1
2
3
4
5
6
7
8
9
1
10
100
1000
10000
Particle Size (microns)

FIGURE 4

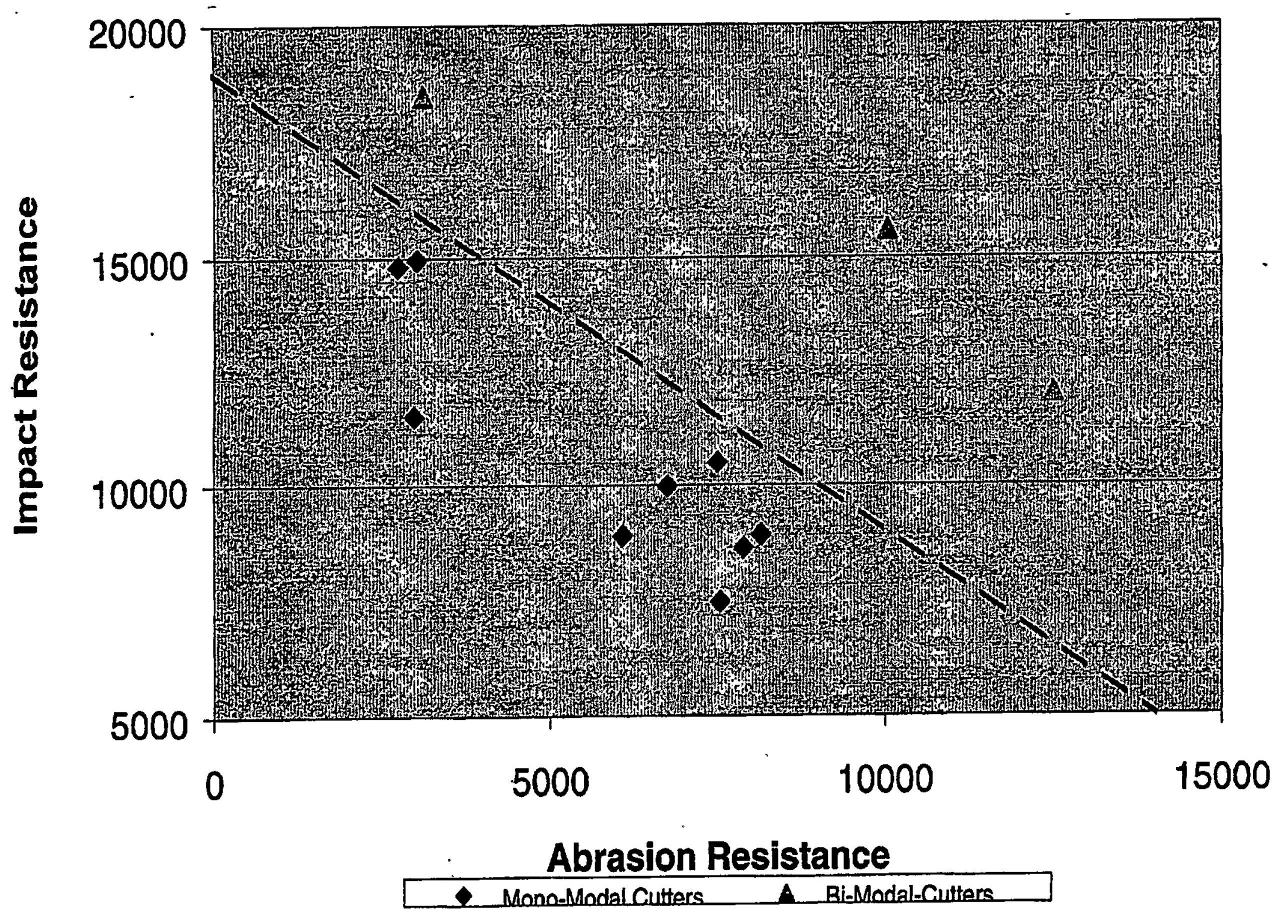

20000
15000
10000
5000
Impact Resistance
0
5000
10000
15000
Abrasion Resistance
Mono-Modal Cutters
Bi-Modal-Cutters

FIGURE 5